

## **Droplet Sampling of an Oil-Based and Two Water-Based Antievaporant Ultra-Low Volume Insecticide Formulations Using Teflon®- and Magnesium Oxide-Coated Slides**

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## SCIENTIFIC NOTE

### DROPLET SAMPLING OF AN OIL-BASED AND TWO WATER-BASED ANTIEVAPORANT ULTRA-LOW VOLUME INSECTICIDE FORMULATIONS USING TEFLON®- AND MAGNESIUM OXIDE-COATED SLIDES

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**ABSTRACT.** We estimated the diameters below which 50% and 90% of the volume of droplets exist ( $Dv_{50}$  and  $Dv_{90}$ , respectively) of 1 oil-based (Permanone 30-30) and 2 water-based (AquaReslin, Aqua-K-Othrine) antievaporant aerosols (with the Film Forming Aqueous Spray Technology [FFAST™]) using Teflon®- and magnesium oxide (MgO)-coated slides and determined whether the aging of the droplets on the slides (up to 60 min) exhibited any significant effect on  $Dv_{50}$  and  $Dv_{90}$  calculations. There were no significant differences in either  $Dv_{50}$  or  $Dv_{90}$  estimates on MgO-coated slides at 0 min and 60 min for all 3 products tested. On Teflon-coated slides, the only product that showed significant difference between 0 min and 60 min in both  $Dv_{50}$  and  $Dv_{90}$  estimates was Aqua-K-Othrine, perhaps due to a difference in formulation components. Specifically, both values corresponding to  $Dv_{50}$  and  $Dv_{90}$  at 60 min decreased by approximately 50% when compared to the values at 0 min. For the other 2 products, AquaReslin and Permanone, aging of droplets on Teflon up to 60 min did not have any significant effect on  $Dv_{50}$  and  $Dv_{90}$  values. To further investigate the behavior of Aqua-K-Othrine droplets on Teflon-coated slides we observed the droplets immediately after spraying and at 10-min intervals under different conditions of temperature and humidity. The majority of the shrinkage occurred within the 1st 10 min after impaction on the slides under all conditions tested. So in most field situations where slides are read several hours or days after collection, this shrinkage would not be observed. The MgO-coated slides should be the preferred field method for sampling droplets of Aqua-K-Othrine with the FFAST antievaporant technology.

**KEY WORDS** Ultra-low volume, droplet sampling, Teflon-coated slides, MgO-coated slides

One of the factors affecting the efficacy of an aerosolized, ultra-low volume (ULV) insecticidal spray (space spray) for targeting flying insects such as mosquitoes is the size of the droplets contained within the aerosol cloud (Mount 1970). One of the most commonly used methods to sample droplets in the field is by impaction on rotating glass microscope slides; this method is simple, affordable, and easy to use.

The coating of the slides used for droplet impaction varies based on the formulation characteristics of the insecticide. For oil-based insecticides, Teflon® coating was found to be a suitable material for droplet impaction (Anderson and Schulte 1971) because oil-based products are relatively nonvolatile and retain their original diameter for extended periods of time (Anderson and Schulte 1971). On the other hand, water-based insecticides are more prone to volatilization/

evaporation and for that reason a magnesium oxide (MgO) slide coating has been used for water-based insecticidal droplets (May 1945, 1950; Rathburn 1970; Matthews 1975) because the impressions that the droplets create upon impaction on the slide are measured, not the actual droplets. These impressions are permanent and allow for a more precise size determination, even though droplets may shrink due to evaporation (Rathburn 1970).

In recent years, a new group of insecticidal formulations were developed as water-based preformed emulsions utilizing an antievaporant technology known as Film Forming Aqueous Spray Technology (FFAST™) to slow droplet evaporation (Groome et al. 1989). In the past, both Teflon and MgO coatings have been used to sample droplets of FFAST products (Brown et al. 1998, 2003; Mickle and Brown 1999, 2000, 2001; Lothrop et al. 2007). However, experiments for direct comparison of these 2 coatings in determining the volume median diameters ( $Dv_{50}$ s) of FFAST® droplets are not known and the appropriateness of Teflon as a FFAST droplet impaction surface has not been investigated in relation to droplet size measurements and shrinkage. Our main objective was to estimate the  $Dv_{50}$  and the  $Dv_{90}$  of 1 oil-based and 2 water-based,

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antievaporant aerosols using Teflon- and MgO-coated slides and to determine whether aging of the droplets on the slides (up to 60 min) has any significant effect on  $Dv_{50}$  and  $Dv_{90}$  calculations.

Three commercially available ULV formulations registered for mosquito adulticiding were used in all experiments: 1 oil-based formulation, Permanone 30-30 (30% permethrin, Bayer Environmental Science, Research Triangle Park, NC), and 2 water-based FFAST formulations, Aqua-Reslin (20% permethrin, Bayer Environmental Science, Research Triangle Park, NC) and Aqua-K-Othrine (2% deltamethrin, Bayer Environmental Science, Lyon, France). The latter product is registered in the European Union but not currently available for use in the USA. All compounds were used neat (undiluted). An earlier version of the Colt 4 portable ULV-generator (London Fogger Inc., Long Lake, MN) was used to atomize all compounds. The generator operated with a flow rate of approximately 60 ml/min.

Battery-powered slide rotators (Compact Aerosol Droplet Sampler Model 312, John W. Hock Company, Gainesville, FL) were used for droplet sampling. Rotators were operated at a speed of 550 rpm and were placed side by side to create a sampling line on a stand approximately 1 m from the ground. In order to create a relatively uniform spray cloud, the handheld generator was facing the rotators and moved at the same height and alongside the sampling line at a constant distance (approximately 2 m). Microscope glass slides ( $25 \times 75 \times 1$  mm) were either coated with Teflon plastic film (BioQuip Products Inc., Rancho Dominguez, CA) or purchased plain (Fisher Scientific Company, Pittsburg, PA) and coated with MgO as described by Chaskopoulou et al. (2011). Three rotators were used containing 1 Teflon- and 1 MgO-coated slide each.

After spraying was completed the rotators, with the slides still attached, were collected and transported to the laboratory. Once in the laboratory the slides were removed from the rotators and placed on the microscope stage individually so the droplets could be measured. The droplets on both coating types were measured immediately after spraying each rotator (within 3 min), and at 60 min postspraying for both coating types. During the 60-min period, the slides were kept on their holding racks in the laboratory where the temperature and humidity remained relatively constant (approximately  $20^{\circ}\text{C}$  and 55% relative humidity [RH]). The droplet sizes were determined under a VanGuard compound microscope (model 14867FLi, Vee Gee Scientific, Kirkland, WA) at  $200\times$  magnification with an ocular micrometer calibrated so that each division measured  $4.5\text{ }\mu\text{m}$ . The diameters of at least 200 droplets were measured per slide for a total of 3 Teflon- and 3 MgO-coated slides per

product tested. For those droplets that did not have a spherical shape, 2 measurements were made corresponding to the narrowest and widest dimension of the droplet. The average of these 2 measurements was used as the droplet diameter.

When estimating the  $Dv_{50}$  and  $Dv_{90}$  values, the correction for volume diameter was done using the diameter (D) value rather than the volume diameter ( $D^3$ ) associated with each droplet, as suggested by Yeomans (1949). A spread factor of 0.61 was used for the 3 compounds on Teflon; spread factors of 0.86, 0.8, and 0.75 were used for droplet sizes  $>20$ , 15–20, and 10–15  $\mu\text{m}$  on MgO coating, respectively (May 1945).

All statistical analyses were performed with the SAS software package (version 8.01; SAS Institute, Cary, NC). A paired *t*-test was performed to determine the effect of droplet aging on the calculation of droplet  $Dv_{50}$  and  $Dv_{90}$  values for droplets sprayed on MgO- and Teflon-coated slides ( $P = 0.05$ ). The  $Dv_{50}$  and  $Dv_{90}$  values of the droplets immediately upon impaction on the slides were calculated and compared with the  $Dv_{50}$  and the  $Dv_{90}$  values of the droplets 60 min postimpact.

The mean  $Dv_{50}$  and  $Dv_{90}$  values of insecticide droplets at 0 and 60 min after impaction on Teflon- and MgO-coated slides were calculated for all 3 products tested. There were no significant differences in either  $Dv_{50}$  or  $Dv_{90}$  estimates on MgO slides at 0 min and 60 min for all 3 products tested (Table 1). On Teflon-coated slides, the only product that showed significant difference between 0 min and 60 min in both  $Dv_{50}$  and  $Dv_{90}$  estimates was Aqua-K-Othrine. Specifically, both values corresponding to  $Dv_{50}$  and  $Dv_{90}$  at 60 min decreased by approximately 50% when compared to the initial readings. For the other 2 products, AquaReslin and Permanone, time did not have any significant effect on  $Dv_{50}$  and  $Dv_{90}$  values.

To further investigate the behavior of Aqua-K-Othrine droplets on Teflon-coated slides we observed the droplets immediately after spraying and at 10-min intervals for 60 min. Spray and slide preparation was repeated as described above, except that this time the effects of 3 different environmental scenarios on droplet behavior were investigated. For the 1st scenario the slides were kept spinning at temperatures  $25\text{--}27^{\circ}\text{C}$  and humidity  $>80\%$  RH, resembling spinning slides in the field during a night spray. The 2nd scenario was identical to the 1st one except that the slides were not spinning. For the 3rd scenario the slides were stationary in their slide racks at conditions of temperatures  $18\text{--}20^{\circ}\text{C}$  and humidity  $<55\%$  RH, resembling a laboratory where slides would normally be stored for droplet measurement after a night-spray trial. In order to observe droplet behavior, the slides were moved immediately to the laboratory after spraying and

Table 1. Mean  $Dv_{50}$  and  $Dv_{90}$  values for Permanone, Aqua-K-Othrine and AquaReslin droplets on MgO- and Teflon®-coated slides immediately (0 min) and 60 min after impaction to slides.<sup>1</sup>

Product	Mean $Dv_{50} \pm SE$				Mean $Dv_{90} \pm SE$			
	Teflon		MgO		Teflon		MgO	
	0 min	60 min	0 min	60 min	0 min	60 min	0 min	60 min
Permanone	5.7 $\pm$ 0.2	5.6 $\pm$ 0.1	6.7 $\pm$ 0.1	7.0 $\pm$ 0.1	11.1 $\pm$ 0.3	11.0 $\pm$ 0.2	13.9 $\pm$ 0.8	14.0 $\pm$ 0.2
Aqua-K-Othrine	8.5 $\pm$ 0.1	4.3 $\pm$ 0.8*	6.2 $\pm$ 0.3	5.9 $\pm$ 0.2	25.8 $\pm$ 5.0	9.4 $\pm$ 1.4*	18.7 $\pm$ 1.0	17.3 $\pm$ 0.4
AquaReslin	7.2 $\pm$ 0.5	6.2 $\pm$ 0.4	7.4 $\pm$ 0.2	7.8 $\pm$ 0.3	18.9 $\pm$ 4.2	11.9 $\pm$ 0.8	14.9 $\pm$ 0.2	15.0 $\pm$ 0.2

<sup>1</sup>  $Dv_{50}$ , volume median diameter;  $Dv_{90}$ , volume 90% diameter; MgO, magnesium oxide.  
\* Mean  $Dv_{50}$  and  $Dv_{90}$  values at 60 min are significantly smaller than corresponding values at 0 min ( $P = 0.05$ ,  $t$ -test).

observed by microscopy (200 $\times$  magnification). Droplet behavior was recorded through time by photographing a set field of approximately 10–20 droplets every 10 min for 60 min. The same field of droplets on each slide was captured every time. This procedure was repeated over 8 different slides and a total of approximately 100 droplets were recorded for each one of the 3 scenarios. The slides were on the microscope stage only long enough to capture the images and were immediately returned to the conditions described above. A digital microscope camera (DCM500, Hangzhou Huaxin IC Technology Inc., Zhejiang, China) with the corresponding computer software (ScopePhoto 3.0 Image Processing, Hangzhou Huaxin IC Technology Inc., Zhejiang, China) was used to capture, manage, and save the image files.

The images of the droplet fields and of a calibrated stage micrometer (1 division = 4.5  $\mu$ m) were printed at the same scale on standard copy paper or transparent acetate sheets (216  $\times$  279 mm), respectively. The stage micrometer on the acetate sheet was used to measure the droplets on the printed images of the droplet fields.

The diameter of Aqua-K-Othrine droplets decreased significantly during all 3 experimental conditions (dry environment/nonspinning slides, moist environment/nonspinning slides, moist environment/spinning slides) (Fig. 1). The majority of the shrinkage occurred during the 1st 10 min after impaction on the slides under all 3 environmental conditions tested.

In previous studies with FFAST antievaporant water-based products some inconsistencies between  $Dv_{50}$  estimated using Teflon- versus MgO-coated slides have been demonstrated. The Teflon slides produced a measured  $Dv_{50}$  that was approximately  $\frac{1}{2}$  the  $Dv_{50}$  measured by using MgO slides (Mickle and Brown 2000, Brown et al. 2003). Mickle and Brown (2001) showed that characterization of their nozzle using a laser wind tunnel in the laboratory yielded a  $Dv_{50}$  nearly 5-fold greater than what was measured in the field using Teflon slides. Findings by these authors suggest that possible evaporation of the aqueous formulation may have resulted into smaller droplets being measured on the Teflon slides.

This study provided direct evidence that FFAST droplets of Aqua-K-Othrine, despite containing antievaporant ingredients, will shrink significantly on Teflon-coated slides within 10 min after impaction and, therefore, MgO slides should be preferred over Teflon. On the other hand, despite

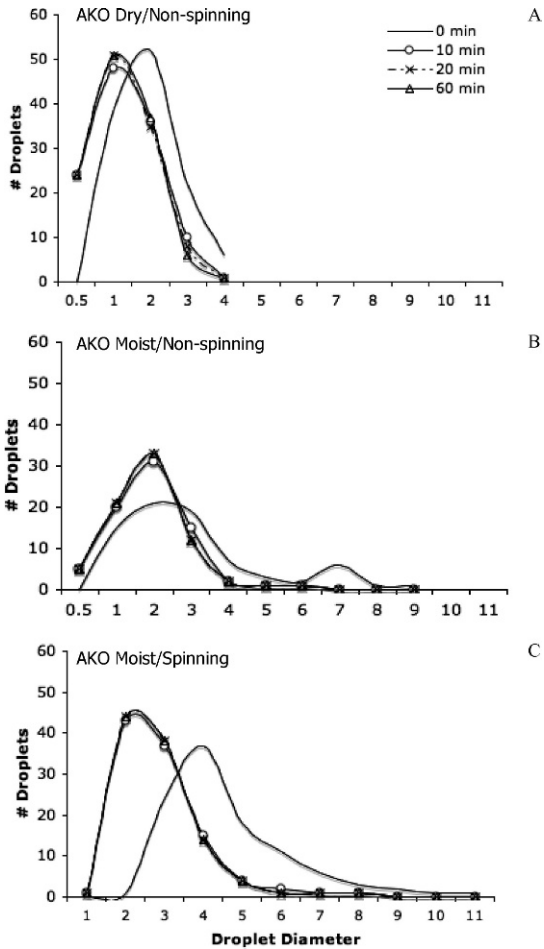


Fig. 1. Droplet diameter (in  $\mu$ m) distribution for Aqua-K-Othrine (AKO) on Teflon®-coated slides at 0, 10, 20, and 60 min. (A) Dry, nonspinning scenario. (B) Moist, nonspinning scenario. (C) Moist, spinning scenario.

losing their spherical shape, AquaReslin droplets on Teflon-coated slides did not shrink significantly within 60 min postspraying. Although both Aqua-K-Othrine and AquaReslin formulations contain the same FFAST antievaporant technology, these formulations contain a combination of ingredients (actives and/or solvents/synergists) with variable physical properties (i.e., vapor pressure) resulting in different evaporation rates.

Magnesium oxide slides also have several disadvantages. Some of the most important disadvantages are the requirement of a long preparation time (Anderson and Schulte 1971) and their high susceptibility to dust and other airborne contaminants, which may leave imprints on the slide surface. Although we currently recommend the use of MgO-coated slides, a new technology is needed for a more accurate droplet sampling of FFAST antievaporant insecticides under field conditions. In the meantime, correction factors adjusted for each FFAST formulation and droplet size will need to be determined to allow for the usage of Teflon-coated slides.

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